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(57) Abstract

High-shear, microcavitation is used to shred and disintegrate lignocellulose-containing biomass. This process is preferably done with the biomass particles dispersed in a slurry created using only water. The treated biomass is particularly suited for use as a particularly digestible feed for ruminant animals.

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TREATMENT METHOD FOR LIGNOCELLULOSIC BIOMASS

This is a continuation-in-part of pending application Serial No. 07/991,745, filed December 17, 1992, by the same Applicant, which application is incorporated by reference in the present application.

The present invention relates to improvements in methods for treating fibrous, lignocellulose-containing biomass to produce animal feed. The purpose of the invention is to produce a particularly digestible feed for ruminant animals from biomass, where the method itself does not hydrolyze the carbohydrates present in the biomass. The invention is particularly directed to the use of a shearing technique, which shatters the fibers in a biomass and renders the carbohydrates more accessible for hydrolysis.

Lignocellulose-containing biomass is made up of carbohydrates, primarily cellulose and hemicellulose, protein, and lignin. Generally, lignocellulosic biomass may consist of about 80% carbohydrates; however, only a relatively small amount of these are available to a ruminant animal. This is thought to be due to the close physical and chemical association between lignin and cellulose or hemicellulose within the cell wall of woody plants. This close association or bonding prevents the ready access of enzymes and other agents to cellulose and hemicellulose for hydrolysis within the animal's stomach or rumen. Additionally, when cellulose molecules exhibit a high degree of crystallinity in their structure they are even more resistant to hydrolysis. Consequently, a great deal of effort has been directed toward finding

methods for the removal of lignin and the disruption and destruction of the crystalline structure of the cellulose molecule.

A key to increasing the sugar yields from lignocellulose-containing biomass, both commercially and to the ruminant animal, is to increase the access to cellulose and hemicellulose by the enzymes or other chemical or biological agents used to hydrolyze them to sugars. Generally, access to cellulose and hemicellulose is increased by increasing the reactive surface area of the biomass. Thus, attempts have been made to destroy the fibrous structure of the biomass and thereby increase the reactive surface area of the biomass. The greater the reactive surface area of the treated biomass, the more access that enzymes or other agents have to cellulose and hemicellulose in the biomass.

Many different techniques have been used to treat lignocellulosic biomass specifically to increase the reactive surface area of the treated biomass. These techniques have resulted in varying degrees of effectiveness.

Concentrated acid has been used to chemically hydrolyze fibrous biomass. Biomass and the acid are combined, forming a broth. The broth is held in a vat at ambient temperature for a period of time sufficient to break down the biomass and hydrolyze the cellulose, hemicellulose, proteins and lignin. While effectively breaking down the biomass and hydrolyzing the cellulose and hemicellulose, this process creates the difficult problem of separating the sugars from the acid. Presently, there is no efficient and economical way to separate the sugars and acid, thus this process is undesirable for a commercial application unless it can be supported by co-product sales. Furthermore, concentrated acid is corrosive and requires extreme care in handling.

The use of concentrated acid is unacceptable for producing a particularly digestible animal feed for at least two reasons: (1) the acid hydrolyzes many of the available sugars from the biomass depriving the animals of their nutritional benefit

and (2) concentrated acids are not acceptable for consumption. There is likely to be some acid remaining post-treatment because as previously stated, acids are difficult and expensive to remove from treated biomass.

Dilute acid has been used in a process to promote the disintegration of fibrous biomass. Biomass and dilute acid are combined, forming a broth. The broth is "cooked" at high temperature causing the hemicellulose to hydrolyze. The acid must then be neutralized and washed out of the mix. Following the removal of the acid, the remaining biomass is treated with high enzyme loadings, in excess of 12 IU's/gram of substrate, to hydrolyze the cellulosic fraction.

Washing the acid out of the mix creates a waste stream that must be treated prior to discharge from the processing facility. "Cooking" at high temperature causes formation of furfural and hydroxymethyl furfural in the sugar component of the mix. Furfural and hydroxymethyl furfural are toxic and must be separated from the desirable sugars. Steps have been taken to minimize furfural production through a two-stage/two-temperature approach. First, a lower temperature hydrolyzes the C_5 sugars, which are removed. Then the remaining fiber is subjected to higher temperatures for decrystallization and delignification.

Separating the acids and other toxic products from the desirable sugars is difficult and expensive, limiting the usefulness of this process. The use of acids also raises environmental issues because it requires the addition of neutralizing base chemicals, thus creating a waste stream.

A method using dilute acids is unacceptable for producing a particularly digestible cattle feed because it also hydrolyzes many of the sugars present in the biomass, thus depriving the animals of their nutritional value. Because the dilute acids are generally neutralized after treatment of the biomass and the acid used is dilute, the concern about residual acid in the feed is not as serious a concern as with the use of concentrated acids.

Steam has been used to disintegrate and defiberize biomass. This is done under high pressure and at a high temperature exploding the fibers within the biomass. Because this procedure must be done at high temperatures, degradation products such as furfural and hydroxymethyl furfural are created. These degradation products are toxic and must be removed prior to fermentation of any sugars produced from the exploded materials. The water wash streams used to remove the degradation products become toxic themselves and must be treated before they can be discharged from the processing facility. Besides the environmental cost, the use of steam is extremely costly because of the energy that is required which is not recoverable, and because of the equipment costs that are required to practice this technique.

Steam also breaks down a portion of the cellulose and hemicellulose present in the biomass making steam explosion an unacceptable method for producing the animal feed of the present invention.

Ammonia has also been used to explode and disintegrate fibrous biomass. This technique, known as AFEX, is performed under high pressure. The pressure and temperature required are dependent upon the substrate being treated. The process is generally performed at temperatures from 50°-110°C. This process does not degrade sugars and suffers from few, if any, adverse environmental effects. Nonetheless, the ammonia must be reclaimed and this entails certain costs. Further details of this technique are described in United States Patent No. 5,037,663 issued to Bruce E. Dale, the subject matter of which is incorporated herein by reference.

While ammonia apparently does not hydrolyze the sugars present, the treated biomass would be unacceptable for consumption unless the ammonia can be completely removed.

Grinding methods have been tried to essentially chop the biomass into pieces small enough for effective sugar hydrolysis. These attempts have not resulted in commercially acceptable sugar yields even though the biomass has been ground to

particle sizes as small as 37 microns. This technique requires no chemicals, so chemical recovery is not a problem. The drawback to grinding is that it is very energy intensive, inefficient, and an expensive means of disintegrating fibrous biomass. Most grinding methods essentially cut the end off of the fiber bundle time after time. The fiber bundle is strongest perpendicular to the fiber axis, and it is in this perpendicular direction that most of the cutting is done. To achieve very small particle sizes, the biomass accordingly must be ground repeatedly. Consequently, such grinding consumes a great deal of energy making grinding simply uneconomical as a means of treatment. Moreover, grinding has been shown to be ineffective as a treatment. The cost of grinding is prohibitive based on the energy consumption alone, not taking into account equipment costs, including the cost of repair and general wear of the machinery.

Strong alkali agents have been used in conjunction with shear forces produced by an extruder device to both chemically and physically disintegrate fibrous biomass. This technique uses a mixture of a strong alkali and a peroxide combined with biomass. The technique requires solids loadings in excess of 30% solids. The alkali-peroxide/biomass broth is exposed to shear forces in an extruder device. The extruder device has a masticatory effect on the biomass, grinding and chewing the particles. The extruder is particularly used for its mixing capabilities, i.e., dispersing the biomass in the alkali-peroxide mix; however, when high-shear mixing is accomplished, structural disintegration occurs. While producing high-shear forces, an extruder functions similar to a grinder. The resulting broth is then held in residence for up to 24 hours to complete the process.

This method requires the use of potentially toxic chemicals. It also may require extended treatment times, up to 24 hours. Recovery of the alkali and peroxide is not necessarily required, but special care must be used in handling these toxic chemicals. This method accordingly is expensive due to the handling costs, and can be inefficient due to the time required for treatment. Further details of this technique are described in United States Patent No. 4,997,488 issued to John M.

Gould and Brian J. Jasberg, the subject matter of which is incorporated herein by reference.

Methods using strong alkali agents suffer from the same problems that methods using strong acids do, at least partial hydrolysis of the sugars present and concern about consumption of feed with residual alkali agents. The method described by Gould and Jasberg neutralizes the alkali in the treated biomass; however, sugar hydrolysis still occurs as a result of the alkali treatment.

High-frequency, rotor-stator devices have been used to aid in disintegrating starch-containing agriculture products like corn and tubers prior to refining the starch to alcohol. Corn and other starch-containing materials, however, have little or no lignin associated with their cell structure; and their cell structure is minimally fibrous when compared with a fibrous lignocellulosic biomass. Consequently, while starch-containing materials have been subject to mechanical breakdown by the use of rotor-stator devices, it has been generally considered that these devices would not be effective in breaking down lignocellulose-containing materials. Furthermore, this process is not used extensively because the process did not prove effective in preparing starch for ethanol production.

Starch-containing materials are not considered appropriate starting materials for the present invention.

The present invention concerns method for the production of a particularly digestible feed for ruminant animals, and the feed produced by the method. The present invention in a general aspect comprises a method of reducing the particle size of a fibrous biomass containing lignocellulose, wherein an aqueous slurry of particles of the biomass is injected cross-currently into a turbulent Couette flow of the slurry. The injected slurry is also preferably in turbulent flow. The Couette flow occurs in a narrow channel or gap between a first stationary surface and a second surface traveling at high speed. The resulting turbulence has proven very effective in not

only reducing the particle size of a fibrous biomass but also shearing or splitting the fibers in such a way that the cellulose and hemicellulose in the biomass becomes especially exposed or susceptible to subsequent hydrolysis, whether occurring commercially or in the stomach or rumen of an animal.

Preferably, the biomass feed is forced to be injected into a succession of the channels or gaps mentioned above. Thus, once a quantity of biomass has been injected into a first channel or gap and mixed with another quantity of biomass traveling in turbulent Couette flow, a first portion of the resulting first mixture is injected into a second such channel to mix with still another quantity of the biomass. At the same time, a second portion of the first mixture continues to flow in the first channel, where it preferably encounters an injection of still another quantity of the biomass.

Preferably, all of the stationary surfaces and rapidly moving surfaces which define the Couette channels or gaps form parts of a stator and a rotor, respectively. Several actual devices which incorporate such stators and rotors are machines currently available under the names SUPRATON™ and DISPAX P/2™. All of these devices have a plurality of generally parallel channels defined between a stator and an adjacent rotor by lands or walls which run around each stator and rotor. Each such land or wall is crenelated to provide a plurality of openings for slurry to either enter into a channel between the lands or to exit from that channel to an adjacent channel. In operation, pulses or slugs of slurry are injected into a given channel to generate pressure pulses in the channel. Then, as the mixed slurry in the channel moves and lines up with an opening leading to the next channel, a slug of the mixture exits the first channel and enters into the next channel where it is injected into a new body of slurry in turbulent Couette flow. This pulsing type of flow from one channel to the next causes the slugs to experience pressure changes which, it is concluded, assist greatly in causing fibers in the slugs to erupt and shear axially. The several mechanisms that occur in this system are not entirely understood, but the results in terms of shredding the fibers in a biomass and improving the hydrolysis of cellulose

components in the biomass have been unexpectedly significant. It has been especially noted that the fibers have been sheared axially as well as transversely, thus opening the fibers to a marked degree. It appears that the pulsing, turbulent nature of the overall flow pattern gives rise not only to high-shearing actions but also to microcavitational effects which promote fiber destruction.

There is minimal grinding in the process of the invention, because there is approximately a 1 mm. space between the rotor and the stator of the rotor-stator devices used in the invention; and particle sizes are generally less than 1 mm. before the slurry is created. High-shearing forces created by the rotor in each ring-shaped path shred the fibrous particles as they pass from one path to the next. As they pass from one ring to another, microcavitation forces help to explode the fibrous particles concurrently with the shearing action.

The particles generated by the several forces vary in size from very fine, about 1-5 microns, to relatively large, about 1-2 mm. The particles also exhibit greatly increased surface area due to the internal disruption of the fibers, allowing easier penetration by the enzymes or other agents involved in sugar hydrolysis.

The invention has particular application in preparing a very desirable animal feed because the feed is more readily and completely digested. The axial shearing and other mechanisms attacking the biomass expose the cellulose and hemicellulose to an unusual degree for ready animal digestion. The feed is especially suitable for ruminant animals, particularly cattle.

Animal feed of the invention is preferably made from fibrous lignocellulose which has a lignin content of less than about 18 percent. Thus, the lignocellulose is derived from grasses, hay, and other non-woody materials such as sugar cane bagasse. Tree bark, wood chips and similar materials generally have lignin contents above 18 percent and are not preferred. In any case, the biomass used in preparing the animal feed should be reduced to a flour, and a slurry then formed using the

flour. The slurry should then be sheared under turbulent Couette flow conditions sufficiently severe to produce longitudinal shearing of the lignocellulosic fibers. This type of shearing has been readily observable when samples of treated biomass have been examined under an electron microscope.

After being sheared, a sufficient amount of the water in the sheared biomass is removed to render the biomass suitable for any particular feeding operation. For example, if the sheared biomass is to be used promptly as in a dairy farm, a feedlot or other relatively large scale operation, the water content in the feed may be relatively high, e.g., as much as 40 percent or even more. On the other hand, if the product is to be bagged, stored or shipped a substantial distance, a relatively low water content will be preferred, i.e., about 10 percent or less. Thus, while simple settling, straining or rough filtration may remove enough water in a situation involving prompt feeding of the product to animals, the use of rotary filters, dryers, filter presses, centrifuges or the like may be required where delayed feeding is the case.

In general, the amount of water to be removed from any particular slurry of sheared biomass should be sufficient to render the product suitable for handling in the animal feeding operation for which the product is intended.

FIG. 1 is a schematic axial view of a portion of a rotor/stator device.

FIG. 2 is a flow diagram depicting an example of the invention carried out in the presence of water alone.

As stated above, the present invention uses high-frequency, rotor-stator shearing technology in the treatment of a lignocellulosic biomass to produce a particularly digestible animal feed. This type of device produces high-shear, microcavitation forces which disintegrate the biomass fed into it. Two commercially produced high-frequency, rotor-stator dispersion devices are the Supraton™ devices

manufactured by Krupp Industrietechnik GmbH and marketed by Dorr-Oliver Deutschland GmbH of Connecticut, and the Dispax™ devices manufactured and marketed by Ika-Works, Inc. of Cincinnati, Ohio.

Preferred raw materials comprise cellulose-containing materials such as grasses, grains, crop residues, hay, sugar cane bagasse, and other types of plant material suitable for animal feed. Preferred is non-woody biomass which pertains to cellulose-containing material having generally a lignin content of 18 percent or less. Thus, biomass in the form of tree bark or wood chips from trees would not be preferred starting materials for the present process. "Hay" as the term is used herein includes those grasses conventionally cultivated for feeding to ruminant animals, such as coastal bermuda grass, alfalfa, and the like, and grasses not conventionally cultivated for feeding to ruminant animals, such as prairie grasses, foxtail, and the like.

To prepare the biomass for shearing, the biomass is first reduced to a flour by grinding, to classify out tramp materials which might damage the rotor-stator shearing device. Grinding to a desired flour may be accomplished in one or more stages. In a preferred embodiment, the milled biomass is ground in a conventional tub grinder to a particle size sufficiently small enough to pass through number 3 mesh sieve. The ground biomass is then fed to a hammermill, or series of hammermills or a disk mill and reduced to a particle size sufficiently small enough to pass through a number 20 mesh sieve.

The ground or milled product is preferably mixed with water to obtain a slurry of a desired solids content. One of the purposes of this portion of the process is to further defibrate the biomass. In a preferred embodiment, the dry powder is fed into a hopper and conveyed to a mixer-grinder-pump and water added to form a slurry having a solids content of up to about 60 percent solids. In a preferred embodiment, the mixer-grinder-pump is a medium shear, rotor-stator device capable of mixing and pumping high solid content slurries. This device further reduces the particle size of

the biomass, wets the particles thoroughly with water, and disperses the particles within the water. Examples of this type of device are the HED™ manufactured and marketed by Ika Works, Inc. of Cincinnati, Ohio and the Gorator™ manufactured by Krupp Industrietechnik GmbH and marketed by Dorr-Oliver Deutschland GmbH of Connecticut.

In a preferred embodiment of the process, the slurry is pumped into a high-frequency, rotor-stator dispersion device where it is subjected to high-shear mixing of two turbulent streams as described earlier. As mentioned earlier, preferred high-frequency, rotor-stator dispersion devices are the Supraton™ and the Dispax™ devices. Referring to Figure 1, a slurry is fed into the high-frequency, rotor-stator device and forced into a chamber 10. Inside the chamber is a series of coaxial meshing rings of teeth. The rings are concentric, radiating out from the center. The rings 12 on the stator are fixed and the rings 14 on the rotor are rotated by a shaft. The teeth 16 on the rotor and the teeth 18 on the stator are closely spaced at close tolerances. The space between the teeth of the rotor and stator is typically about 1 mm.

Adjacent pairs of teeth are separated by gaps 20 and 22. The tooth and gap size determine the coarseness of the machine, i.e., a coarse tool has fewer teeth with larger gaps between adjacent teeth when compared with a medium or fine tool. Both the Supraton™ and Dispax™ allow the use of coarse, medium, and fine toothed rings in the same device, or the devices can have all coarse, all medium, or all fine toothed rings in the chamber so that the machines may be used in series.

As the slurry is pumped under pressure into the chamber 10 by the mixer-grinder-pump, it encounters each concentric layer of teeth as the slurry is forced laterally. This lateral force is created by the pressure on the slurry as it is pumped into the chamber by the mixer-grinder-pump and by the centrifugal force created by the spinning rotor. The slurry passes through the gaps between the teeth as the rotor spins past the gaps in the stator. Flow is most pronounced when the gaps 22 between

the rotor teeth align with the gaps 20 in the stator. The result is a pulsing flow with a rapid succession of compressive and decompressive forces. The lignocellulosic material in the slurry is subjected to these repeated forces, as the centrifugal force accelerates it through the gaps toward the outer edge of the chamber. The repeated compressive and decompressive forces create microcavities in the slurry with extremely intensive energy zones. The lignocellulosic fibers are ripped apart by this intensive energy.

As the lignocellulosic particles pass outward through the various gaps, they also come in contact with the teeth. Some grinding of the particles occurs due to such contact. The grinding effects are relatively small, however, when compared with the combined effects of shear and microcavitation.

Grinding typically cuts, slices, and dices fibrous material perpendicular to the fiber bundle, producing a more spherical type of particle. Shear in combination with microcavitation, on the other hand, tends to shatter the material, that is, it rips the fibers apart from the inside-out forming irregularly shaped particles. Examination of these particles show them to have been "cut" both perpendicular to the fiber axis and longitudinally along the fiber axis. The effect on the fibers is to shatter their structure, disrupting the lignin bonding to cellulose. Shattering the individual particles of the biomass results in a particulate substrate which is more readily penetrated by the desired enzymes used in sugar hydrolysis, and thus more easily digested by the ruminant animal.

As previously stated, high-frequency, rotor-stator dispersion devices may have differently configured rings or "tools" within the chamber. These tools, for example, may vary in the gap size between the teeth on the rings. With a larger gap size, the resulting material is more coarse than with a smaller gap size. As stated earlier, these tools can be varied within one device to contain coarse, medium, and fine rings in the chamber of the device. Likewise, a device may contain rings of the same

rating so that the devices can be staged. This capability is important for use in a continuous process.

Processing a fibrous biomass through one or more of the high frequency, rotor-stator dispersion devices renders the fibrous material especially well suited for subsequent hydrolysis of the cellulose components. The fibers have been thoroughly shredded, and the associated cellulose material is readily available for hydrolytic attack. Thus, as animal feed, the treated biomass is "pre-chewed" and prepared for enzymatic hydrolysis and direct microbial conversion to produce C_5 and C_6 sugars.

FIG. 2 illustrates an embodiment of the invention which is preferred for use on grasses and other biomass of low lignin content, preferably 18% or less. In this embodiment a fibrous lignocellulosic biomass is ground in stage 30 to a dry flour. This flour is combined with water in a mixer-grinder-pump in stage 32 to form a slurry. The resulting slurry is sheared in a series of rotor-stator devices, passing through a coarse device 34, then a medium device 36, and finally a fine device 38.

As explained earlier, the complex forces created by each rotor-stator device shatter, shred, and disintegrate the lignocellulose particles in the slurry. The net effect has been unexpected, literally exploding the fibers and ripping them apart. Electron microscope studies show that the effect begins internally within the fibers. The resulting particles are very small and due to the internal disruption of the fiber structure, the reactive surface area of the fibers is greatly increased.

In addition to being an excellent source of C_5 and C_6 sugars for alcohol fermentation, treated biomass is an excellent carbohydrate feed source for ruminant animals. The nutrients contained in the biomass, in the form of carbohydrates, become accessible to the enzymes and microbes residing in the stomachs of ruminant animals, such as cattle. Thus, a cow gets the same benefits that accrue to a fermentation process; the cellulose is more accessible to its naturally occurring

enzymes and indwelling microbes. Consequently, the animal may make more complete and efficient use of the biomass eaten.

Untreated grasses tend to be excreted only partially digested because the fibrous structure of the biomass and lignin-cellulose bonding make access to the cellulose and hemicellulose present by the indwelling enzymes and microbes very difficult. Through the use of the high-frequency, rotor-stator dispersion device, the fibrous structure of the biomass and the lignin-cellulose bonding are disrupted making the cellulose more susceptible to enzymatic and microbial attack. Thus, treated biomass is made more digestible than untreated grass. As a result of treating the biomass, 70-100% of the digestible matter present in the biomass is made available to the animal. More of the nutrients from the grass are available to the animal, and consequently less of the feed is excreted partially digested. The result is more efficient use of feed for the rancher or feed lot operator and increased nutrient intake from less volume of feed for the ruminant animal.

Following the shearing stages, much of the water is removed from biomass in drying stage 40. Water removal may be done any number of ways, for example the treated biomass may be centrifuged or filter pressed. The retentate may be fed directly without further treatment as the process does not use any chemicals other than water. Alternatively, the retentate may be pelletized or cubed either with or without being supplemented with various feed additives or binders. Corn and soybeans are examples of feed additives that may be used to supplement the treated biomass before feeding.

The foregoing was for purposes of illustrating the invention. Those skilled in the art will recognize that various modifications can be made to the invention, and all such modifications are incorporated within the spirit and scope of the invention.

CLAIMS:

1. A method of making an animal feed from hay, which comprises:

a) providing in flour form a fibrous lignocellulosic biomass containing less than about 18 percent lignin;
b) mixing the flour with water to form a slurry;
c) shearing the slurry under Couette flow conditions to longitudinally shear fibers in the biomass; and
d) removing water from the slurry of the sheared biomass to produce an animal feed.

2. An animal feed prepared by the method of claim 1.

3. A method for preparing feed for ruminant animals from fibrous lignocellulosic biomass, comprising:

a) reducing the particle size of a fibrous lignocellulosic biomass containing less than about 18% lignin to produce a flour;
b) mixing the flour with water to produce a slurry ranging up to 60% solids;
c) shearing the slurry under conditions to cavitate the slurry and produce axially extending shearing in the biomass fibers; and
d) removing water from the sheared slurry to produce a feed for ruminant animals.

4. An animal feed prepared by the method of claim 3.

5. A method for preparing feed for ruminant animals from fibrous lignocellulosic biomass, comprising:

a) mixing a fibrous lignocellulosic flour with water to produce a slurry;
b) shredding the slurry using high-shear, microcavitation; and
c) removing water from the shredded slurry to produce a feed for ruminant animals.

6. An animal feed prepared by the method of claim 5.

7. The method of claim 5, wherein the shredding produces particles ranging in size from about 1 to 5 microns to about 1 to 2 millimeters.

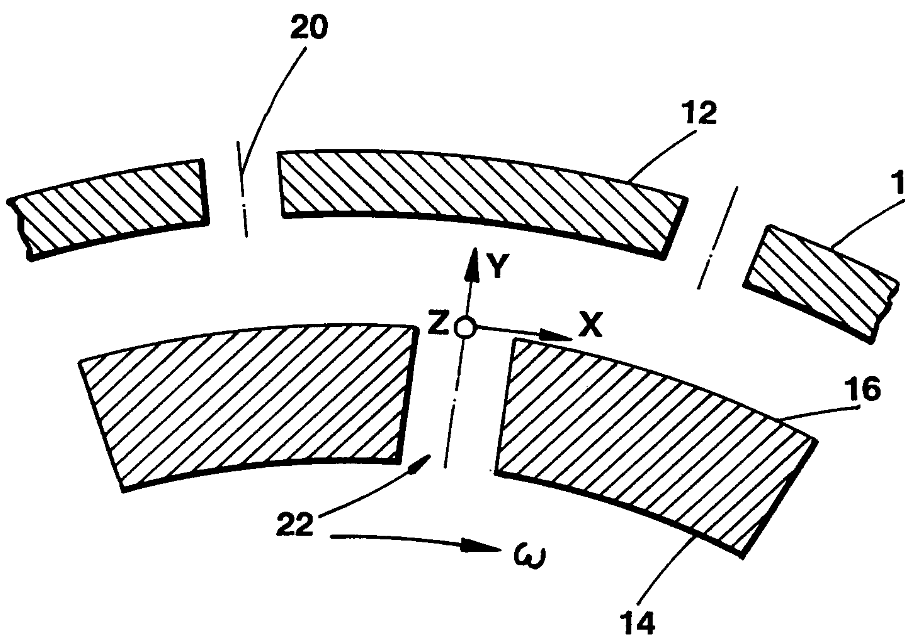
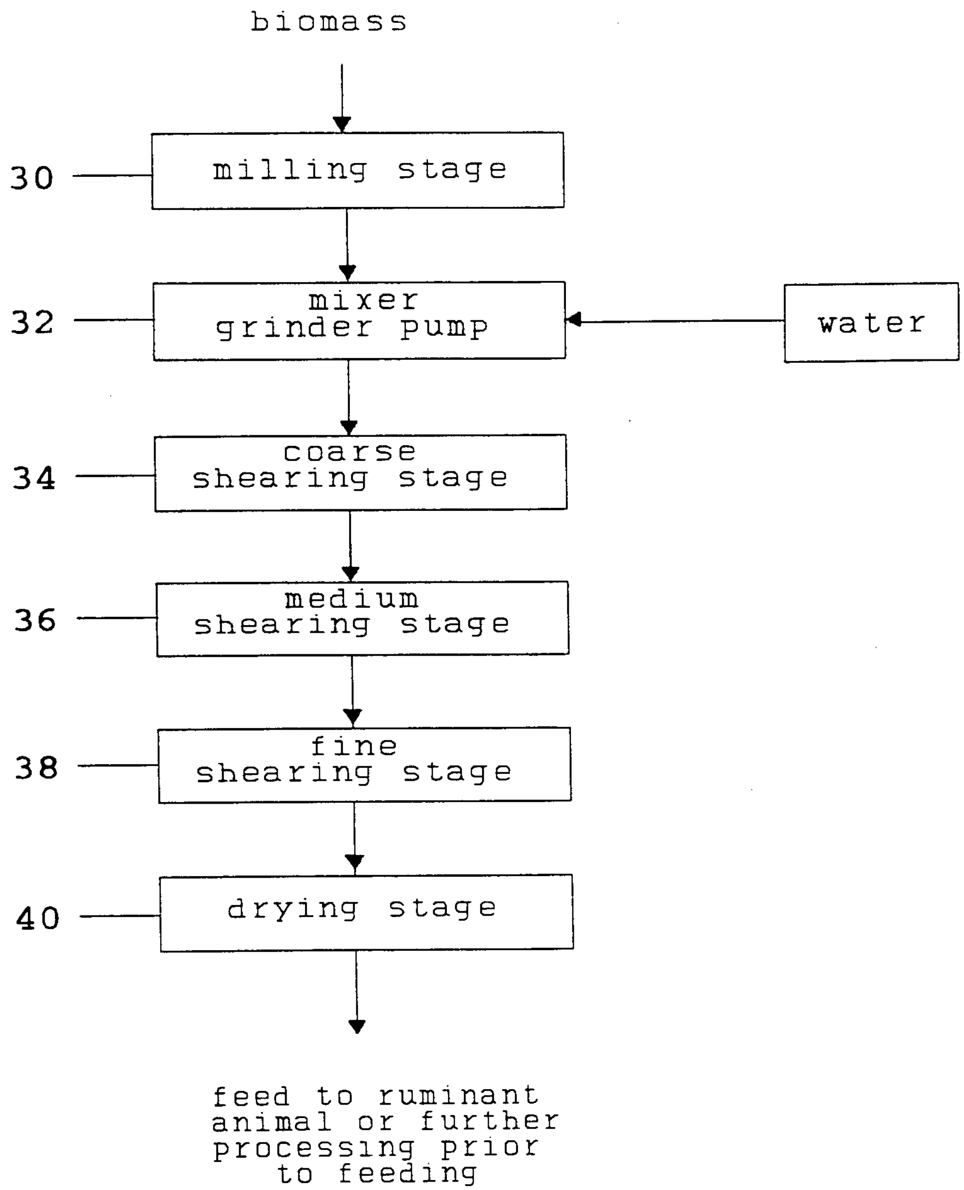


Fig. 1

Fig. 2


biomass
30
milling stage
32
mixer grinder pump
water
34
coarse shearing stage
36
medium shearing stage
38
fine shearing stage
40
drying stage
feed to ruminant animal or further processing prior to feeding

INTERNATIONAL SEARCH REPORT

International Application No
PCT/US 94/09112

A. CLASSIFICATION OF SUBJECT MATTER

A 23 K 1/14,A 23 K 1/18,C 13 K 1/02

According to International Patent Classification (IPC) or to both national classification and IPC 6

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

A 23 K,C 13 K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X,P	WO, A, 94/13 838 (COLORADO STATE UNIVERSITY) 23 June 1994 (23.06.94), claim 1, pages 6,7. --	1-7
A	DE, A, 3 211 346 (SUPRATON F.J.ZUCKER) 29 September 1983 (29.09.83), claims. --	1-7
A	DE, A, 3 545 746 (FRIED.KRUPP GMBH) 25 June 1987 (25.06.87), claims. --	1-7
A	DE, A, 2 827 923 (SIMMERING-GRAZ-PAUKER AG) 19 April 1979 (19.04.79), claims.	1-7

[X] Further documents are listed in the continuation of box C.

[ ] Patent family members are listed in annex.

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Date of the actual completion of the international search	Date of mailing of the international search report
30 November 1994	20.12.94

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax (+31-70) 340-3016	IRMLER e.h.

International Application No
PCT/US 94/09112

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Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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ANHANG

zum internationalen Recherchenbericht über die internationale Patentanmeldung Nr.

ANNEX

to the International Search Report to the International Patent Application No.

ANNEXE

au rapport de recherche international relatif à la demande de brevet international n°

PCT/US 94/09112 SAE 96376

In diesem Anhang sind die Mitglieder der Patentfamilien der im obengenannten internationalen Recherchenbericht angeführten Patentdokumente angegeben. Diese Angaben dienen nur zur Unterrichtung und erfolgen ohne Gewähr.

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